AMMRC CTR 77-17

DEVELOPMENT OF OXYNITRIDE MATERIALS FOR CERAMIC TOOL BITS

June 1977

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

SIALON ceramic compositions were prepared by hot pressing mixtures of silicon nitride, silica and aluminum nitride. Dense microstructures were obtained and X-ray diffraction analyses were performed. Transverse bend strength measurements were performed at room temperature and 1300°C for the various compositions.

Cutting tool evaluation tests were performed against hardened tool steel, cast iron and a specialty alloy René 41.

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I. INTRODUCTION

Oxynitride ceramic materials of the SIALON variety are the subject of a number of investigations concerned with their preparation and evaluation as materials for advanced turbine engine components. Jack[1] has reported the stability of alumina-rich SIALON compositions in contact with iron-rich melts and there are indications that a number of Japanese scientists are investigating their potential as cutting tool materials.[2]

Recent work[3] has also demonstrated that alumina containing SIALON compositions possess mechanical bend strength properties rivaling those of the best hot pressed fine-grained pure alumina compositions, e.g., $\sim$50,000 psi at 1200°C.

The fine-grain size character of these SIALON materials coupled with good mechanical properties and good corrosion resistance enhances their prospects as cutting tool materials.

It is recognized, however, that SIALON compositions are likely to be multi-phase, in view of recent findings.[4,5] The major phase resulting from the reaction of Al_2O_3 and Si_3N_4, for example, is a solid solution based on an expanded β-Si_3N_4 structure and labeled β'. The solubility limit at 1750°C of Al_2O_3 in Si_3N_4 has been estimated to be about 67 percent. However, several investigators who have reacted mixtures of Al_2O_3 and Si_3N_4 report the presence of other phases beside the β' phase.[6,7]

Also, recent reports[8,9,10] of phase equilibrium studies in the system Si_3N_4-Al_2O_3-AlN-SiO_2 have shown that single phase β' solid solutions do not exist for any appreciable extent between Si_3N_4 and Al_2O_3. Rather extensive β' solid solutions are found along the line extending from Si_3N_4 toward the composition $AlN \cdot Al_2O_3$.

This project was concerned with the development and evaluation of a family of SIALON (oxynitride) ceramics for use as ceramic tool bit materials.

Three compositions were selected to cover a range of alumina content and they are represented by the following formulas:

(1)	$Si_{15}Al_2O_3N_{20}$	or $(Si_3N_4)_5Al_2O_3$	13 wt. % Al_2O_3
(2)	$Si_6Al_2O_3N_8$	or $(Si_3N_4)_2Al_2O_3$	27 wt. % Al_2O_3
(3)	$Si_3Al_2O_3N_4$	or $(Si_3N_4)Al_2O_3$	42 wt. % Al_2O_3

Raw materials included AME controlled phase grade silicon nitride, 5 µm particle size fused silica from Harbison Walker Co., and fine particle aluminum nitride from Shieldalloy Corp.

Alumina contents beyond these levels were thought likely to show significant multi-phase content and remain to be investigated because of the added complexity.

Billets of these compositions were fabricated into 2" x 2" x ½" thick shapes and the resulting material characterized by metallographic and X-ray diffraction procedures.

Four-point transverse bend tests were performed to establish a property-microstructure composition relationship, and selected compositions were evaluated as cutting tool insert materials.

II. RESULTS AND DISCUSSION

Billets based on compositions described above were fabricated by hot pressing mixtures of silicon nitride, aluminum nitride and silica in graphite dies in a flowing atmosphere of N_2 gas at temperatures ranging from 1550 - 1750°C. Above 1700°C some decomposition was observed in the form of sample bloating and temperatures in subsequent trials were maintained below this level.

Table I provides data on the sialon billet fabrication conditions and

Table I - Sialon Billet Fabrication Conditions

Run No.	Material Composition	Temp. °C	Pressure psi	Time (min.)	Billet Density, gm/cc
1911	No. 1 $(Si_3N_4)_5\ Al_2O_3$	1550	3000	120	2.6
1913	No. 1 $(Si_3N_4)_5\ Al_2O_3$	1650	3000	120	2.8
1913	Repress	1750	3000	120	Bloated specimen Low density
1914	No. 1 $(Si_3N_4)_5\ Al_2O_3$ plus 2 wt. % MgO	1700	3000	120	3.10
1915	No. 1 $(Si_3N_4)_5\ Al_2O_3$ plus 2 wt. % MgO	1600	3000	120	3.19
1916	No. 2 $(Si_3N_4)_2\ Al_2O_3$	1650	3000	120	2.91
1920	No. 3 $(Si_3N_4)\ Al_2O_3$	1750	3000	120	Bloated Low Density
1921	No. 1 $(Si_3N_4)_5\ Al_2O_3$ plus 2 wt. % MgO	1550	3000	120	3.16
1922	No. 2 $(Si_3N_4)_2\ Al_2O_3$ plus 1 wt. % MgO	1600	3000	120	3.15
1923	No. 3 $(Si_3N_4)\ Al_2O_3$ No MgO	1600	3000	120	3.13
1924	No. 1 $(Si_3N_4)_5\ Al_2O_3$ plus 1 wt. % MgO	1600	3000	120	3.16

Table I concluded

Run No.	Material Composition	Temp. ^{o}C	Pressure psi	Time (min.)	Billet Density, gm/cc
1925	No. 1 $(Si_3N_4)_5$ Al_2O_3 plus 1 wt. % MgO	1600	3000	120	3.16
1926	No. 3 (Si_3N_4) Al_2O_3 No MgO	1600	3000	120	3.12
1928	No. 1 $(Si_3N_4)_5$ Al_2O_3 plus 1 wt. % MgO	1600	3000	120	3.14
1930	No. 3 (Si_3N_4) Al_2O_3 plus 1 wt. % MgO	1600	3000	120	3.13
1934	No. 3 (Si_3N_4) Al_2O_3 plus 1 wt. % MgO	1600	3000	120	3.11

results of densification. To facilitate densification for Compositions 1 and 2, $(Si_3N_4)_5Al_2O_3$ and $(Si_3N_4)_2Al_2O_3$, respectively, a 1 wt. % MgO additive was found to be very effective. Otherwise, decomposition at higher temperatures precluded the attainment of high densification without an additive.

Composition No. 3, on the other hand, did not require an additive for identical process conditions as indicated by Run No. 1923. An MgO additive of 1% did not significantly alter the degree of densification as revealed in runs 1930 and 1934. The action of the MgO in the SIALON compositions is probably similar to its action in silicon nitride in providing a low temperature liquid phase which enhanced densification by a solution reprecipitation mechanism.

Microstructure and X-ray examinations were conducted on the fabricated billets and Figures 1, 2, and 3 show representative microphotographs of polished sections of samples of the three selected compositions. Figures 1 and 2 (Compositions 1 and 2) show essentially completely dense microstructures with some observable second phase constituent. Figure 3 (Composition 3 - Billet 1923) shows a small amount of residual porosity as well as second phase constituent. This latter composition contained no MgO additive as a densification aid since sufficiently high densities were attained without it.

X-ray diffraction analyses were performed and revealed the presence of two major phases, i.e., β' phase and α phase of silicon nitride. Crystalline silica and/or silicon oxynitride phases (Si_2ON_2) were not detected. The possibility of an AlN polytype exists. Two high intensity lines were found as an indication of the presence of aluminum nitride, but the absence of a third intense "d" line at 2.372 probably precludes its actual presence. Other lines observed which could not be indexed included:

Plate 6129-6 250X

Figure 1. Photomicrograph of Polished Section of Billet No. 1928 (Comp. 1)

Plate 6129-4 250X

Figure 2. Photomicrograph of Polished Section of Billet No. 1922 (Comp. 2)

Plate 6129-5 250X

Figure 3. Photomicrograph of Polished Section of Billet No. 1923 (Comp. 3)

d Spacing	Intensity I/I_{max}
2.747	7
2.238	14
2.110	17
1.622	7
1.608	11
1.584	17
1.419	9

Bend test bars were prepared and measurements were performed at room and elevated temperatures for the three compositions. The results are provided in Table II. These results show that Composition 1 is strongest at room temperature by a reasonably large margin, e.g., about 20% over Compositions 2 and 3. On the other hand, the elevated temperature strength for Compositions 1 and 2 are comparable but somewhat lower than that for Composition 3, which may be a function of the MgO additive in Compositions 1 and 2 providing more liquid phase.

For the bar size and span to thickness ratio observed, the strength properties of these compositions are somewhat lower at room temperature than a 99% pure fine grained (1-3 μm size) hot pressed alumina tool. However, at 1300°C, the strength properties observed for these SIALON compositions appear definitely superior to hot pressed alumina.

Samples of these compositions were prepared into cutting tool inserts (SNG 432 designation) and evaluated at Metcut Corp. in Cincinnati, Ohio in a series of performance tests.

Evaluations were performed with hardened steel (50 R_c), cast iron and a specialty alloy René 41. The results for the first two cases are plotted in Figures 4 and 5 and show that the SIALON compositions are not very effective for use against a hardened steel. In addition, a hot pressed silicon nitride composition (Norton HS130) fared no better. A standard hot pressed alumina base cutting tool, Type NPCA2 based on an 80% Al_2O_3 and 20% TiC composition ,

Table II - Bend Strength Results for Sialon Compositions

(Sample size 1 3/4" x .2" x .1")

Composition & Billet No.	Temp.	Four Point Bend Strength Kpsi		Elastic Modulus psi
Composition No. 1				
1928-1	R.T.	58.9		
1928-2	"	72.0	Av. 58.2	42.8×10^6
1928-3	"	45.2		
1928-4	"	60.5		
Composition No. 1				
1928-5	1300°C	42.4		
1928-6	"	37.8	Av. 39.6	
1928-7	"	36.2		
1928-8	"	41.5		
Composition No. 2				
1922-1	R.T.	52.4		
1922-2	"	42.7	Av. 48.2	41.9×10^6
1922-3	"	50.0		
1922-4	"	47.5		
Composition No. 2				
1922-5	1300°C	38.5		
1922-6	"	42.0	Av. 39.0	
1922-7	"	37.2		
1922-8	"	38.1		
Composition No. 3				
1923-1	R.T.	44.7		
1923-2	"	46.1	Av. 45.5	38.5×10^6
1923-3	"	48.5		
1923-4	"	42.5		
Composition No. 3				
1923-5	1300°C	45.5		
1923-6	"	42.9	Av. 44.1	
1923-7	"	41.8		
1923-8	"	46.1		

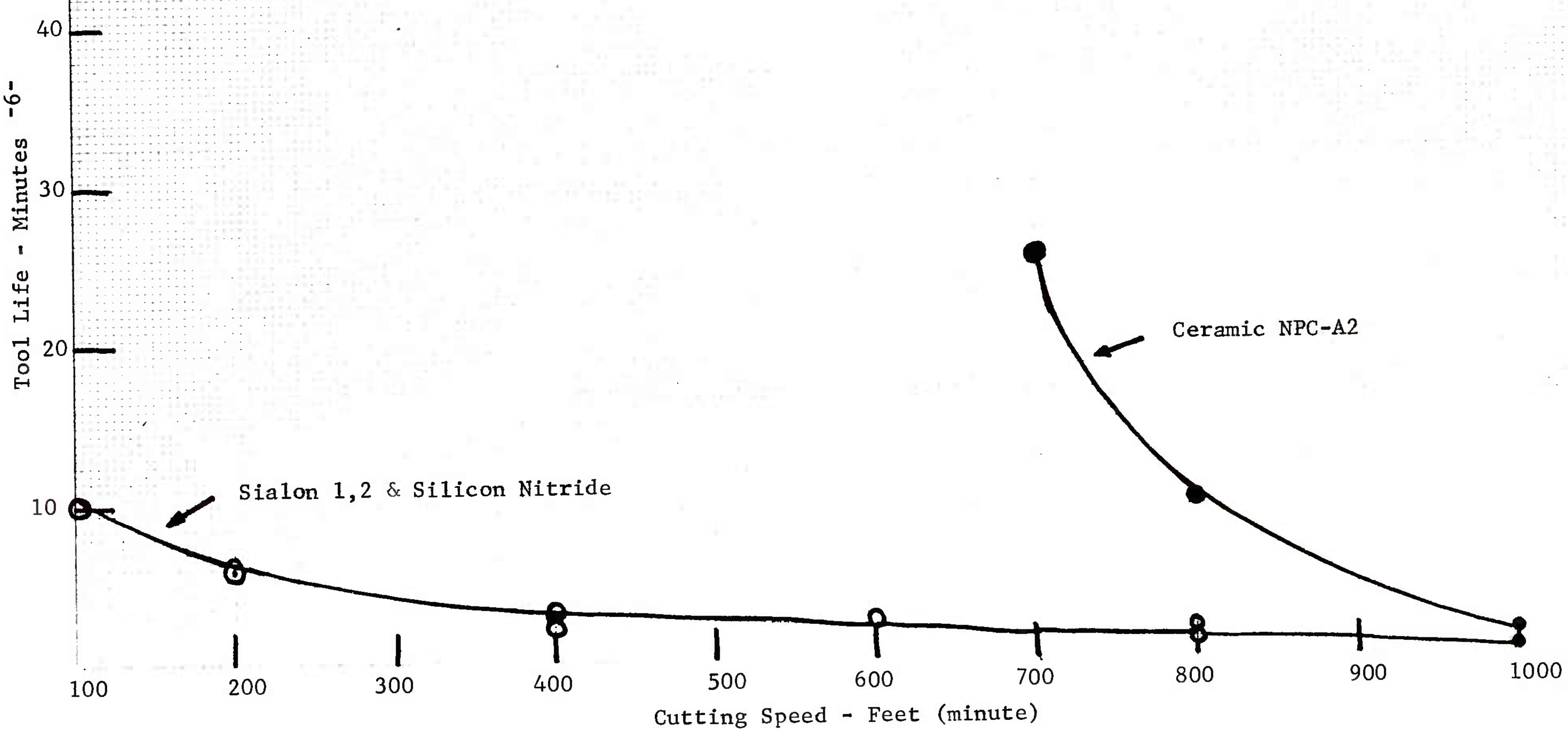
Figure 4. CUTTING TOOL EVALUATION AGAINST 4340 STEEL

Turning 4340 Steel Quenched and Tempered 50 R_c

Tool Material: Sialon Comp. 1 & 2, Hot Pressed Silicon Nitride
(Alumina Ceramic NPC A-2 in SNG-432 Configuration)

Feed: .005 in/rev.
Depth of Cut: .050"
Cutting Fluid: Dry

Tool Life End Point: .015" Uniform Wear
.030" Localized Wear or Chipping



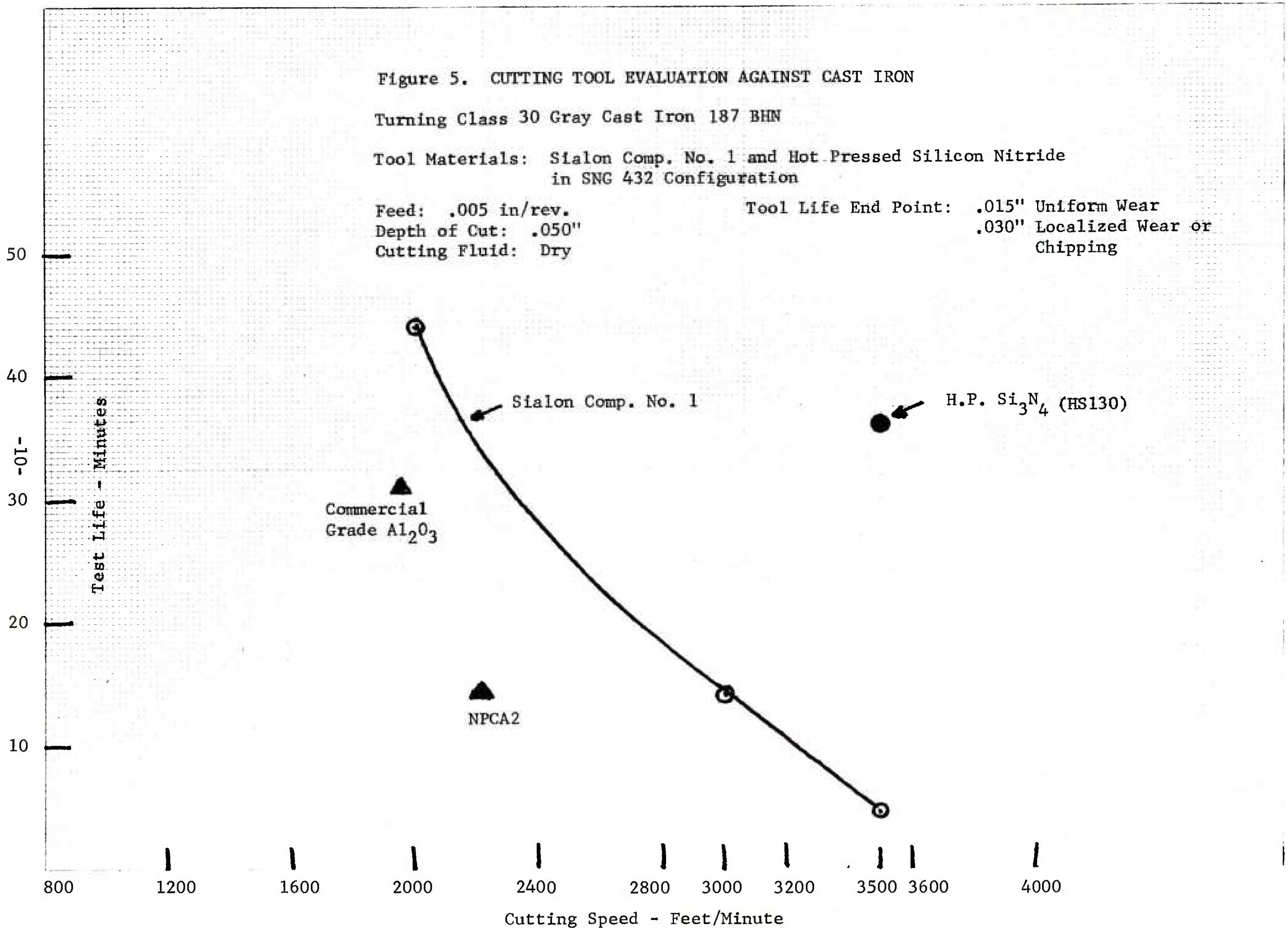


Figure 5. CUTTING TOOL EVALUATION AGAINST CAST IRON
Turning Class 30 Gray Cast Iron 187 BHN
Tool Materials: Sialon Comp. No. 1 and Hot Pressed Silicon Nitride in SNG 432 Configuration
Feed: .005 in/rev.
Depth of Cut: .050"
Cutting Fluid: Dry
Tool Life End Point: .015" Uniform Wear
.030" Localized Wear or Chipping
Sialon Comp. No. 1
H.P. Si_3N_4 (HS130)
Commercial Grade Al_2O_3
NPCA2
Test Life - Minutes
10
20
30
40
50
800
1200
1600
2000
2400
2800
3000
3200
3500
3600
4000
Cutting Speed - Feet/Minute

was superior by a factor of about 10 to 1 at a cutting speed of 800 surface feet per minute.

Against cast iron, however, (Figure 5) SIALON and silicon nitride (HS 130) compositions were very effective. The performance life of the hot pressed silicon nitride at 3500 sfm was exceptional. These lifetimes are considered superior to hot pressed alumina base (Al_2O_3/TiC (Avco Act I)) compositions, although no tests were actually performed. A data point for hot pressed alumina is provided in Figure 5 based on earlier work on part of Contractor. Also a data point for NPCA2 is provided on the plot. The behavior is considered peculiar in view of the relatively poor performance against silicon nitride tests against the smooth finish of the hard steel. It is considered that cutting tool-steel stock reactions are possible, e.g., iron silicide formation at the interface leading to adherence and chip formation.

In the case of cast iron where an irregular and intermittent surface is presented to the cutting tool, impact resistance may be the important parameter and silicon nitride compositions are known to excel in this connection.

Testing with René 41 alloy did not reveal very promising behavior for either the hot pressed silicon nitride or the SIALON composition selected (No. 1), although the latter was superior. In both instances, low cutting speeds had to be employed and the results are tabulated below.

Table III. René 41 Alloy-Cutting Tool Evaluation

Tool Composition	Cutting Speed (sfm)	Tool Life (min.)
SIALON 1	100	1 2/3
SIALON 1	50	6
H.P. Silicon Nitride	100	½
H.P. Silicon Nitride	50	3/4

Again early chipping failures were observed that may have been reaction related.

III. CONCLUSIONS AND RECOMMENDATIONS

In the limited evaluation conducted, hot pressed, dense SIALON and silicon nitride compositions have shown excellent performance behavior as a tool material for cast iron. Their performance against hardened steel and the specialty alloy René 41 was not particularly promising, however.

Failure mechanisms may be chemical reaction related, but this remains to be determined by reaction product studies.

It is recommended that a complete assessment of these nitride compositions against cast irons and other tool compositions be determined to establish the potential for their usage in production machining operations.

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